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## (54) OLEFIN POLYMERIC FIBRES

(71) We, MONTECATINI EDISON S.p.A., a Body Corporate organised and existing under the laws of Italy, of 31 Foro Buonaparte, Milan, Italy, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

The invention relates to olefin polymeric fibres suitable for use in pulp in the manufacture of paper, generally without cutting or further preparatory treatment.

The invention provides a process of producing such fibres which comprises ejecting a solution of an olefin polymer as a jet and hitting the jet of solution with a jet of fluid. The solution of the olefin polymer is preferably at a temperature higher than the boiling temperature of the solvent in normal conditions, and at autogenous or like elevated pressure. The ejection is preferably into the ambient atmosphere which is at lower pressure and so allows the ejected solution to expand. The jet of fluid is preferably at high speed and at a temperature lower than that of the olefin polymer solution. The jets are preferably inclined to each other at an angle, generally of from 30° to 90°, particularly from 40° to 90°.

Suitable olefin polymers include (crystalline) polyolefins obtained from monomers of the formula:  $R-CH=CH_2$ , wherein R is an alkyl or aryl group, or a hydrogen atom, such as polyethylene, polypropylene, polypentene-1, poly-4-methylpentene-1, polystyrene or a copolymer obtained from a mixture of such monomers, possibly in admixture with each other. Linear polyethylenes obtained using supported Ziegler catalysts have proved particularly interesting, as have polypropylenes consisting essentially of isotactic macromolecules with a high index of isotacticity obtained using Ziegler-Natta catalysts, and mixtures of such

polyolefins with minor proportions of copolymerized units of other monomers such as polyvinyl chloride, polyvinyl acetate, polymethyl methacrylate, polyamides, polyoxymethylene, and cellulose acetate. For preparing fibres endowed with a high cohesive power, there may be used polyolefins modified by the presence therein of polar groups.

It is advisable, although not indispensable, that the solvent in the olefin polymer solution should have a boiling temperature lower than the melting temperatures of the polymer. In general there may be used all those solvents, liquid or gaseous under normal conditions, that give homogeneous solutions of the polymer. Suitable solvents include aliphatic hydrocarbons such as *n*- or *iso*-butane, pentane, hexane, heptane, and octane; cycloaliphatic hydrocarbons such as cyclohexane; aromatic hydrocarbons, such as benzene, toluene and xylene; and chlorine containing hydrocarbons such as chlorobenzene, trichloroethylene, tetrachloroethylene, and trichlorofluoromethane.

A wide range of concentrations of polymeric solutions may be used, depending on molecular weight and on type of polymer. In general there may be used solutions containing from 1 to 700 grams per litre (g/l) of polymer, preferably from 50 to 400 g/l. Solutions having, under operational conditions, absolute viscosities like those at 130°C in a solution obtained by dissolving in 1 litre of hexane 100 g of polyethylene having an intrinsic viscosity  $[\eta]$  in tetralin at 135°C = 0.9 are most preferred.

Pigments, fillers, stabilizing agents, anti-static agents and/or other substances for modifying the surface properties of the fibres may be added to the olefin polymer solution. Particularly helpful is the addition to the solution of surfactants, which gives fibres readily dispersible in water. This dispersability is an important factor for their utilization in the preparation of paper. The lack of hydro-

philic properties of olefin polymeric fibres makes such dispersion difficult. On the other hand, the addition to the water of surfactant compounds before or during the dispersion involves certain drawbacks, such as the formation of a foam that causes stratification of mixes of olefin polymeric fibres with cellulose fibres. The addition of a wetting agent directly to the olefin polymeric solution before its extrusion avoids this difficulty.

The wetting or surfactant agent used, should of course be uniformly soluble or miscible with the solvent and the olefin polymer. It may be of the anionic, cationic, non-ionic, or amphoteric type. Amongst suitable surfactants of the anionic type there may be cited soaps of fatty acids, soaps of naphthenic acids, salts of sulphuric acid esters, alkali metal sulphonates, alkyl esters of phosphorus or phosphoric acid, salts of alkyl-phosphoric esters, and sodium salts of sulphuric esters of alkylphenol-polyethyleneglycol. Suitable surfactants of the cationic type include quaternary ammonium alkyl compounds, aliphatic amines, basic salts of alkylpyridinium or of alkylpicolinium and alkylbenzimidazole derivatives. Suitable amphoteric surfactants include compounds of the betaine and of the sulfo-betaine type, and sulphuric and phosphoric esters. Suitable non-ionic surfactants include polyoxyethylene-alkyl esters and ethers, polyoxyethylene-alkyl-aryl esters, esters of fatty acids with higher alcohols, polyoxyethylene-alkyl amines, alkanol-amides of fatty acids, and block polyoxyethylene-polyoxy-propylene and polyoxyethylene-alkylthioether copolymers.

The surfactant should remain incorporated in the fibres, or at least adhering to the surface of the fibre as long as possible. To this end, the surfactant should have a boiling temperature higher than that of the solution at the moment of its extrusion. By means of a suitable choice of surfactant, it is thus possible greatly to improve the ability of the fibres to form a suspension in the aqueous medium. The antistatic properties of the fibres, and the surface properties of paper sheets prepared from them, can similarly be improved.

The quantity of surfactant to be added to the olefin polymer solution should be greater than 0.05%, preferably 0.1% or more, by weight calculated on the olefin polymer. For reasons of economy, the maximum quantity of surfactant that may be added may be less than 5% by weight calculated on the polymer. Greater quantities will not yield appreciable advantages as far as the dispersion of the fibres in water is concerned. The surfactant may be dissolved or dispersed in the organic solvent before, after or during the dissolving of the olefin polymer in the solvent.

The speed at which the solution of olefin polymer is ejected may be from 1,000 to 200,000 metres per hour (m/h), but is prefer-

ably from 1,500 and 50,000 m/h. The solution to be ejected should preferably have a temperature at least 40 degrees C, but more preferably 60 degrees C higher than the boiling temperature of the solvent under normal conditions. The fluid nozzle is preferably positioned in such a way that the fluid hits the olefin polymer solution at a distance from its exit nozzle. The distance depends mainly on the ejection speed of the solution, but is preferably from 1.5 mm to 15 mm.

As hitting fluid there may be used any liquid, gaseous or vapourized substance which is inert and under operational conditions, does not dissolve the olefin polymer, and is not miscible to any appreciable extent with the solvent of the olefin polymeric solution. Water and steam have proved particularly suitable for the purpose in as much as, in comparison with other usable fluids, they have the advantage of humidifying the fibres, and thereby facilitating their gathering while eliminating the danger of conflagration due to the static electricity with which the fibres tend to become charged. However, nitrogen, oxygen, carbon dioxide, air or a combustion gas, finely divided water or a mixture of two or more thereof may alternatively be used.

The speed of the hitting fluid is very important to the viscosity of the solution and the speed with which this solution is ejected through the nozzle. It has been found that the best operational conditions are obtained when operating at an impact speed of the fluid of from 200 and 600 metres per second (m/s).

The angle between the fluid jet and that of the solution is preferably from 50° to 55° for nitrogen, from 80° to 85° for carbon dioxide or steam, and from 40° to 60° for oxygen. The hitting fluid may be in the form of a mass which is geometrically co-axial with the olefin polymeric solution itself. Very uniformly shaped fibres are thus obtained which is particularly helpful for making paper of good surface properties.

The invention is illustrated by way of example in the drawings of which:

Figure 1 is a diagram of a plant in which the process of the invention can be performed;

Figure 2 shows in greater detail certain parts of Figure 1;

Figure 3 shows an alternative arrangement of certain parts of Figure 1; and

Figure 4 is an enlargement (54x) of fibres produced according to the invention.

With reference to Figure 1, an olefin polymer solution is fed through a pipe 1 into an autoclave 2 equipped with a stirrer 3, and ejected as a jet through a nozzle 6. Hitting fluid is fed through a pipe 4 and a nozzle 5. The fibres fall into a collection vessel 7.

In Figure 2, the nozzles 5 and 6 are shown in greater detail. In Figure 3, a duct 11 for polymeric solution is arranged coaxially inside

a duct 12 for hitting fluid. The ducts 11, 12 respectively end with nozzles 13, 14. A frustoconical chamber 15 allows the polymeric solution to expand before being hit. Terminal zones 16, 17 of the ducts 1, 2 are so shaped that the axis of the space 18 between them is inclined at an angle  $\alpha$  to that of the nozzle 13.

The following Examples illustrate the invention:

#### EXAMPLE 1.

Into a 50 litre stainless steel autoclave, fitted with a jacket and a blade stirrer having a maximum speed of 300 revolutions per minute (rpm), there were charged 30 litres of technical hexane and 2 kg of polyethylene obtained using a supported Ziegler catalyst, modified with propylene, and having melt index=0.021; intrinsic viscosity in tetralin at 135° C=3.0; Specific Gravity=0.950; number of methyl groups per 100 carbon atoms=0.83; and melt temperature (by DSC)=132° C. The autoclave was heated by circulating steam in the jacket until it contained a solution at pressure=2.2 kg/sq. cm; and temperature=108° C. The solution was ejected from the autoclave under these conditions into the atmosphere through a circular nozzle of 2 mm diameter at a flow rate of 50 litres per hour (l/h), and hit at a distance of 2.5 mm from the nozzle by a steam jet at a speed upon impact of 470 m/s from a 4 mm diameter nozzle arranged at a right angle to the solution nozzle.

A mixture of steam, fibres and organic solvent was obtained, and conveyed through a duct to a filter in which the moist fibres were separated from the mixture. The organic solvent content of the fibres was less than 0.3%. (All % herein are by weight). Visual analysis under a VISOPAN microscope, showed the product to consist of about 50% of single fibres having a length of from 1 to 10 mm and a diameter of from 5 to 50  $\mu$ , and of about 50% of single flat fibres rolled up on themselves and having a length of from 1 to 10 mm, a width of from 100 to 500  $\mu$  and a thickness of from 5 to 50  $\mu$ . From surface measurements with a PERKIN ELMER Sorptometer of absorption of nitrogen, the product as a whole was shown to have a surface area below 1 sq m/g.

150 g of the fibres obtained were admixed to 350 g of RAUMA cellulose in 25 litres of water. The mixture was refined in a Lorentzen-Wettres hollander. From time to time samples of pulp were withdrawn which, after dilution, were made into sheets using a laboratory sheet-forming machine. The properties of the sheets thus obtained are in Table 1, in which the properties have been determined according to the ATICELCA (Associazione Tecnici Italiani Celulosa e Carta) rules, as in the other Tables hereof.

#### Example 2.

Into the autoclave of Example 1, there were charged 30 litres of technical hexane and 3 kg of polyethylene as in Example 1. By introducing steam into the heating jacket, there was produced in the autoclave a solution at pressure=2.4 kg/sq cm; and temperature=104° C. Through the nozzle of Example 1, the solution was ejected from the autoclave at a flow rate of 45 l/h into the ambient atmosphere and hit at a distance of 2.5 mm from the nozzle exit by a steam jet at a speed upon impact of 470 m/s. The product gathered on the filter consisted of about 50% of single fibres having a length of from 1 to 20 mm and a diameter of from 5 to 50  $\mu$ , and of about 50% of single flat fibres rolled upon themselves and having a length of from 1 to 20 mm, a width of from 100 to 500  $\mu$ , and a thickness of 5 to 50  $\mu$  with a surface area below 1 sq m/g.

150 g of this fibrous product were admixed to 350 g of RAUMA cellulose in 25 litres of water, and this mixture was used for preparing sheets, following the procedure described in Example 1. The properties of the sheets are in Table 1.

#### Example 3.

In the autoclave of Example 1, there was prepared a solution of 30 litres of technical hexane and 2.5 kg of polyethylene as in Example 1. By heating, a solution was produced in the autoclave at pressure=2.2 kg/sq cm; and temperature=103° C. Through the nozzle device of Example 1, the solution was ejected from the autoclave at a flow rate of 60 l/h, and hit at a distance of 2 mm from the nozzle exit with a steam jet at a speed upon impact of 470 m/s.

The product gathered consisted of about 80% of single fibres from 1 to 5 mm long with a diameter of from 5 to 20  $\mu$ , and of about 20% of single flat fibres rolled up on themselves and having a length of from 1 to 5 mm, a width of from 50 to 100  $\mu$ , and a thickness of from 5 to 20  $\mu$ , and had a surface area of about 1 sq m/g. A series of mixtures was prepared, each consisting of 120 g of the above product and of 280 g of cellulose respectively of the RAUMA, Birch, Modo and Kraft types in a 20 litres of water. ("Modo" is a trade mark). These mixtures were made into sheets as in Example 1. The properties of the sheets are in Table 2.

#### Example 4.

In the autoclave of Example 1, a pressure=14.5 kg/sq cm; and a temperature=134° C were maintained with a solution consisting of 30 litres of trichloro-fluoromethane and 3 kg of polyethylene obtained using a supported Zeigler catalyst and having melt index=18.5; intrinsic viscosity in tetralin at 135° C=0.9; density=0.952 g/cc; number

of methyl groups per 100 carbon atoms = 0.65; and melt temperature (by DSC) = 130° C. Through the nozzle of Example 1, the solution was ejected from the autoclave into the ambient atmosphere at a flow rate of 90 l/h, and hit at a distance of 3 mm from the exit with a steam jet at an impact speed of 470 m/s. There was obtained a fibrous product consisting of about 80% of single fibres with a length of from 1 to 3 mm and a diameter of from 5 to 15  $\mu$ , and of about 20% of single flat fibres rolled up on themselves and having a length of between 1 and 5 mm, a width of between 50 and 100  $\mu$  and a thickness of between 5 and 15  $\mu$ , and having a surface area of 2 sq m/g. Starting from a mixture of 150 g of this product and 250 g of Rauma cellulose in 25 litres of water, and proceeding as in Example 1, sheets were prepared and their properties are in Table 1.

#### Example 5.

In the autoclave of Example 1, a pressure = 5.1 kg/sq cm; and a temperature = 137° C were maintained with a solution consisting of 30 litres of technical hexane and of 3 kg of polyethylene obtained with a Ziegler supported catalyst and having melt index = 18; intrinsic viscosity in tetralin at 135° C = 0.9; density = 0.962 g/cc; number of methyl groups per 100 carbon atoms = 0.21 and melt temperature (by DSC) of 131.5° C. Through the nozzle of Example 1, but with the nozzles arranged at an angle of 85°, the solution was ejected from the autoclave into the ambient atmosphere at a flow rate of 95 l/h, and hit at a distance of 3 mm from the nozzle exit by a carbon dioxide jet at room temperature and at a speed upon impact of 220 m/s. The product obtained consisted of about 90% of single fibres with a length of from 2 to 4 mm and a diameter of about 5  $\mu$ , and of about 10% of flat fibres having a length of from 2 to 4 mm, a width of about 50  $\mu$  and a thickness of about 5  $\mu$ , and had a surface area of 3.5 sq m/g.

#### Example 6.

In the autoclave of Example 1, a pressure = 4.8 kg/sq cm; and a temperature = 135° C were maintained with a solution consisting of 30 litres of technical hexane and 3.5 kg of polyethylene obtained using a Ziegler supported catalyst and having melt index = 49; intrinsic viscosity in tetralin at 135° C = 0.9; density = 0.952 g/cc; number of methyl groups per 100 carbon atoms = 0.28 and melt temperature (by DSC) = 131° C. The nozzle of Example 1 was used but the angle between the two nozzles was 85°. The solution was ejected into the ambient atmosphere and hit at a distance of 2.5 mm from the nozzle by a steam jet. The flow rate of the ejected solution = 55 l/h; the impact speed of the steam = 320 m/s.

The fibrous product obtained consisted of about 70% of single fibres with a length of from 2 to 5 mm and a diameter of from 1 to 5  $\mu$ , and of about 30% of single flat fibres having a length of from 2 to 5 mm, a width of from 50 to 100  $\mu$  and a thickness of from 1 to 5  $\mu$ , and had a surface area of about 3 sq m/g. Starting from a mixture of 150 g of this product with 350 g of cellulose (60% Birch, 20% Modo and 20% Kraft), and proceeding as in Example 1, four sheets were prepared having the properties in Table 3.

#### Example 7.

In the autoclave of Example 1, a pressure = 5.9 kg/sq cm; and a temperature = 160° C were maintained with a solution consisting of 30 litres of technical hexane and of 4.8 kg of polyethylene as in Example 6. The nozzles were as in Example 1 but arranged at an angle of 80°. The solution was ejected into the ambient atmosphere and hit at a distance of 3.5 mm from the nozzle by a steam jet. The flow rate of the solution = 125 l/h; and the impact speed of steam jet = 320 m/s. The product obtained consisted of about 80% of fibres with a length of from 2 to 5 mm and a diameter of from 1 to 5  $\mu$ , and of about 20% of flat fibres having a length of from 2 to 5 mm, a diameter of from 50 to 100  $\mu$  and a thickness of from 1 to 5  $\mu$ , and had a surface area of 5 sq m/g. Starting from a mixture of 150 g of this product with 350 g of cellulose (60% Birch, 20% Modo and 20% Kraft), and proceeding as in Example 1, three sheets were prepared whose properties are in Table 3.

#### Example 8.

In the autoclave of Example 1, a pressure = 5.9 kg/sq cm; and a temperature = 155° C were maintained with a solution consisting of 30 litres of technical hexane and of 1.8 kg of polyethylene as in Example 6. The nozzles as in Example 1 were used, but arranged at an angle of 50°. The solution was ejected into the ambient atmosphere and hit at a distance of 3.5 mm from the nozzle by an oxygen jet at room temperature. The flow rate of solution = 120 l/h; and the impact speed of oxygen jet = 470 m/s. The product consisted almost entirely of single fibres having a length of about 4 to 5 mm and a diameter of about 5  $\mu$  and had a surface area of 11 sq m/g. The organic solvent content of the fibres was less than 0.3%.

#### Example 9.

In the autoclave of Example 1, a pressure = 5.5 kg/sq cm; and a temperature = 145° C were maintained with a solution consisting of 35 litres of technical hexane and 3 kg of polyethylene obtained using a supported Ziegler catalyst and having melt index = 13.6; intrinsic viscosity in tetralin at 135° C = 1; den-

sity=0.9543 g/cc; number of methyl groups per 100 carbon atoms=0.6; and melt temperature (DSC) (differential scanning calorimeter)=130° C. The nozzles as in Example 1 were used and the solution ejected into the ambient atmosphere, and hit at a distance of 3 mm from the nozzle by an oxygen jet at room temperature. The flow rate of the ejected solution=100 l/h; the impact speed of the oxygen jet=470 m/s.

The product consisted of about 80% of fibres from 1 to 3 mm long and with a diameter of from 5 to 20  $\mu$ , and of about 20% of flat fibres from 1 to 3 mm long, width from 50 to 100  $\mu$  and a thickness of from 5 to 20  $\mu$ , and had a surface area of 4 sq m/g. Starting from a mixture of 150 g of this product with 350 g of RAUMA cellulose, and proceeding as in Example 1, four sheets were prepared whose properties are in Table 4.

#### Example 10.

In the autoclave of Example 1, a pressure=5.4 kg/sq cm; and a temperature=142° C were maintained with a solution consisting of 30 litres of technical hexane, 2.4 kg of polyethylene as in Example 9 and of 0.6 kg of an ethylene-ethyl acrylate copolymer (Zetafin 80 of Dow Chemical) (Zetafin is a trade mark). The nozzles as in Example 1 were used and the solution ejected into the ambient atmosphere and hit at a distance of 3 mm from the nozzle exit by a steam jet. The flow rate of the ejected solution=100 l/h; and the impact speed of steam jet=470 m/s. The product consisted of about 80% of fibres from 1 to 3 mm long and from 5 to 20  $\mu$  in diameter, and of about 20% of flat fibres from 1 to 3 mm long, from 50 to 100  $\mu$  wide and having a thickness of from 5 to 20  $\mu$ ; and had a surface area of 4 sq m/g and a specific Gravity of 0.9450. Starting from a mixture of 150 g of this product with 350 g of RAUMA cellulose, and proceeding as in Example 1, four sheets were prepared whose properties are in Table 4.

#### Example 11.

In the autoclave of Example 1, a pressure=5.4 kg/sq cm; and a temperature=139° C were maintained with a solution consisting of 35 litres of technical hexane, 2.55 kg of polyethylene as in Example 9 and 0.45 kg of polyvinyl chloride (K value=45). Nozzles as in Example 1 were used, and the solution ejected into the ambient atmosphere and hit, at a distance of 4 mm from the nozzle by a steam jet. The flow rate of the ejected solution=110 l/h; and the impact speed of steam jet=470 m/s. The product consisted of about 85% of fibres from 1 to 3 mm long and from 5 to 15  $\mu$  in diameter, and of about 15% of flat fibres from 1 to 3 mm long, from 50 to 100  $\mu$  wide and with a thickness of from

5 to 15  $\mu$ ; and had a surface area of 5.5 sq m/g. The Specific Gravity of the fibres was 0.9905. The organic solvent content of the fibres was less than 0.3%. Starting from a mixture of 150 g of this product with 350 g of RAUMA cellulose, and proceeding as in Example 1, four sheets were prepared whose properties are in Table 4. The preparation of the sheets was facilitated by the greater density of the fibrils.

#### Example 12.

In the autoclave of Example 1, a pressure=3.4 kg/sq cm; and a temperature=124° C were maintained with a solution consisting of 35 litres of technical hexane, 3 kg of the polyethylene as in Example 9, and 3% of titanium dioxide calculated on the polyethylene. The nozzles of Example 1 were used but with the nozzles arranged at an angle of 50° and the solution was ejected into the ambient atmosphere and hit, at a distance of 5 mm from the ejecting nozzle by a nitrogen jet at room temperature. The flow rate of the ejected solution=95 l/h; and the impact speed of the nitrogen jet=470 m/s. The product consisted of about 80% of fibres from 2 to 4 mm long and from 1 to 5  $\mu$  in diameter, and of 20% of flat fibres from 2 to 4 mm long, from 50 to 100  $\mu$  wide and from 1 to 5  $\mu$  in diameter, and had a surface area of 3.5 sq m/g and a Specific Gravity of 0.98. Starting from a mixture of 150 g of this product with 350 g of RAUMA cellulose, and proceeding as in Example 1, four sheets were prepared whose properties are in Table 4.

#### Example 13.

In the autoclave of Example 1, a pressure=5.5 kg/sq cm; and a temperature=163° C were maintained with a solution consisting of 30 litres of technical hexane, 2.1 kg of a polypropylene of a high isotactic index obtained using a Ziegler catalyst and having a melt index=6.7; Specific Gravity=0.9085; melt temperature (DSC)=165° C. The nozzles of Example 1 were used but at an angle of 70° and the solution was ejected into the ambient atmosphere and hit at a distance of 7 mm from the ejecting nozzle by a steam jet. The flow rate of the ejected solution=40 l/h; and the impact speed of steam jet=470 m/s. The product consisted almost completely of fibres from 1 to 5 mm long and from 5 to 20  $\mu$  in diameter and had a surface area of 7 sq m/g. Starting from a mixture of 150 g of this product with 350 g of cellulose (60% Birch, 20% Modo and 20% Kraft), and proceeding as in Example 1, a sheet was prepared whose properties are in Table 5.

#### Example 14.

In the autoclave of Example 1, a pres-

sure = 4.5 kg/sq cm; and a temperature = 155° C were maintained with a solution consisting of 30 litres of technical hexane and 3 kg of low-density polyethylene having melt index = 4.6; Specific Gravity = 0.9235; melt temperature (DSC) = 118° C. Using the nozzles of Example 1, but with the nozzles arranged at an angle of 60°, the solution was ejected into the ambient atmosphere and hit, at a distance of 7 mm from the ejecting nozzle, by nitrogen at room temperature. The flow rate of the ejected solution = 30 l/h; and the impact speed of the nitrogen = 470 m/s. The product consisted of fibres from 1 to 3 mm long and from 5 to 15  $\mu$  in diameter, and had a surface area of 13 sq m/g and an organic solvent content of the fibres of less than 0.3%. Starting with a mixture of 150 g of this product obtained with a 350 g of cellulose (60% Birch, 20% Modo and 20% Kraft), and proceeding as in Example 1, a sheet was prepared whose properties are in Table 5.

#### Example 15.

In the autoclave of Example 1, a pressure = 3.0 kg/sq cm; and a temperature = 140° C were maintained with a solution consisting of 30 litres of technical hexane, and 2.1 kg of a polyethylene obtained using a Ziegler unsupported catalyst, and having melt index = 0.47; Specific Gravity = 0.9603; number of methyl groups per 100 carbon atoms above 0.1, and melt temperature = 134° C. Using the nozzles of Example 1 but at an angle of 70° the solution was ejected into the ambient atmosphere and hit, at a distance of 5 mm from the nozzle, by a carbon dioxide jet at room temperature. The flow rate of the ejected solution = 95 l/h; and the impact speed of carbon dioxide jet = 320 m/s. The product consisted of about 70% of fibres from 1 to 10 mm long and from 5 to 20  $\mu$  in diameter, and of about 30% of flat fibres from 1 to 10 mm long, from 50 to 100  $\mu$  wide and from 5 to 20  $\mu$  thick, and had a surface area of about 2 sq m/g. Starting from a mixture of 150 g of this product with 350 g of cellulose (60% Birch, 20% Modo and 20% Kraft), and proceeding as in Example 1, a sheet was prepared whose properties are in Table 5.

#### Example 16.

In the autoclave of Example 1, a solution consisting of 30 litres of technical hexane and 3 kg of the polyethylene of Example 5 was prepared. The solution in the autoclave was heated to a pressure = 5.6 kg/sq cm; and a temperature = 132° C. Using the nozzles of Example 1 the solution was ejected into the atmosphere and hit, at a distance of 5 mm from the ejecting nozzle, by a steam jet. The flow rate of the solution = 90 l/h; and the impact speed of steam jet = 470 m/s. The

product consisted of about 90% of single fibres from 1 to 3 mm long and with a diameter of from 5 to 15  $\mu$  and of about 10% of flat fibres rolled up on themselves and having a length of from 1 to 3 mm, a width of about 50  $\mu$  and a thickness of from 5 to 15  $\mu$ , and had a surface area of 2.5 sq m/g. The preparation was repeated six times so as to get 15 kg of product. 12 kg of product were admixed with 27.6 kg of cellulose (60% Birch, 20% Modo, and 20% Kraft) and 9.5 kg of kaolin in about 1200 litres of water. The mixture was continuously refined in a conical refiner up to 36° SR (Shopper-Reigler), and mixed with 0.08 kg of an optical bleach (Calcofluor 4 MB), 0.8 kg of glue (Aquapel (Trade Mark) 360 XZ), and 1.2 kg of co-adjuvant (Kymene (Trade Mark) 557). To this mixture there was added water to double the volume, and the whole was transferred into a feed vat of a continuous drum-type machine having a useful width of about 55 cm. There were thus prepared 40 kg of paper whose properties are in Table 6 (Test B) together with those obtained in a comparative test using only 40 kg of cellulose (60% Birch, 20% Modo and 20% Kraft) (Test A). Part of the paper obtained from the pulp containing polyethylene fibres had been calendered between two rolls maintained at about 140° C. The results of this operation also are in Table 6 (Test C).

The following Examples illustrate the use of surfactants in the polyolefin solution:

#### Example 17.

Into a 150 litre autoclave fitted with a heating sleeve and a blade stirrer, there were charged 6 kg of polyethylene having melt index = 4.1; Specific Gravity = 0.9633; number of methyl groups per 100 carbon atoms = 0.1; melting temperature (DSC) = 133° C together with 30 g of an ethoxylated stearylamine surfactant and 70 litres of technical hexane. By heating with oil, the autoclave was raised to a temperature = 150° C; total internal pressure = 7 kg/sq cm; nitrogen overpressure = 1.6 kg/sq cm. By means of a pipe sheathed with a steam-heated sleeve, the solution was conveyed to a nozzle having a diameter of 2 mm, ejected through the nozzle into the outer ambient atmosphere, and hit, at a distance of 2.5 mm from the ejection nozzle, by a nitrogen jet at room temperature flowing from a 4 mm diameter nozzle at an angle of 50° to the first nozzle. The temperature of the solution at the nozzle = 158° C; the pressure of the solution at the nozzle = 7.2 kg/sq cm; the pressure of the nitrogen at the nozzle = 21 kg/sq cm; the flow rate of the solution = 100 kg/h; the impact speed of the nitrogen = 320 m/s. The product that was gathered, examined under a Visopan (Riechert) microscope, and appeared to consist of 80% of fibres from 2 to 5 mm long

with a diameter of 1 to 5  $\mu$ , of 30% of flat fibres from 2 to 5 mm long, from 20 to 50  $\mu$  wide and from 1 to 5  $\mu$  thick, and to contain less than 0.3% of solvent. The surface area of the product, measured with a Perkin-Elmer Sorptometer, was 2.9 sq m/g. 150 g of these fibres were admixed with 350 g of cellulose (60% Husum Birch, 20% Husum Kraft and 20% Modo Crown) in 25 litres of water. The mixture dispersed immediately was refined in a Lorentzen-Wettres hollander and, after dilution, used for producing sheets in a laboratory sheet-forming machine. The properties of the sheets are in Table 7.

#### Example 18.

Into the autoclave of Example 17 there were charged 6 kg of polyethylene as in Example 17, 30 g of a nonylphenol ethoxylate surfactant (molar ratio nonylphenol/ethylene oxide=1:6), and 70 litres of technical hexane. By heating with oil, there were established in the autoclave a temperature=155° C; a total pressure=8.2 kg/sq cm; and a nitrogen overpressure=1.6 kg/sq cm. Using the same procedures and equipment as in Example 17, but with the nozzles arranged at an angle of 60°, the solution was ejected into the ambient atmosphere, and hit, at a distance of 5 mm from the ejection nozzle, by a nitrogen current at room temperature. The temperature of solution at the nozzle=175° C; the diameter of solution-ejecting nozzle=2 mm; the flow rate of solution=108 kg/h; the pressure of solution at the nozzle=9 kg/sq cm; the diameter of nitrogen-ejecting nozzle=4 mm; the pressure of nitrogen at the nozzle=20 kg/sq cm; the impact speed of the nitrogen=370 m/s. The product was gathered and shown to consist of 80% of fibres from 1 to 3 mm long and having a diameter of from 1 to 10  $\mu$ , and of 20% of flat fibres from 1 to 3 mm long, from 20 to 50  $\mu$  wide and from 1 to 10  $\mu$  thick, and to have a surface area of 2.5 sq m/g. 150 g of these fibres were admixed to 350 g of cellulose (60% Husum Birch, 20% Husum Kraft and 20% Modo Crown) in 25 litres of water. The fibres immediately dispersed completely in the water. Starting from this aqueous mixture and proceeding as in Example 1, sheets were prepared having properties as in Table 7.

#### Example 19.

Into the autoclave of Example 17 there were charged 6 kg of polyethylene as in Example 17, 30 g of nonylphenol-ethoxylate surfactant (1 mol of nonylphenol per 7.5 mols of ethylene oxide), and 70 litres of heptane. By heating with oil, there were established inside the autoclave a temperature=165° C; a total pressure=7.0 kg/sq cm; and a nitrogen overpressure=2 kg/sq cm. Using the procedures and equipment of Example 17, but with the nozzles arranged at an angle of

85°, the polymeric solution was ejected into the ambient atmosphere and hit, at a distance of 5 mm from the ejection nozzle, with a flow of carbon dioxide at room temperature. The temperature of the solution at the nozzle=172° C; the diameter of the solution-ejecting nozzle=2 mm, the flow rate of the solution=100 kg/h; the pressure of the solution at the nozzle=9.0 kg/sq cm; the diameter of the carbon dioxide-ejecting nozzle=4 mm; the pressure of the carbon dioxide at the nozzle=19 kg/sq cm; and the impact speed of the carbon dioxide=300 m/s. The product consisted almost totally of fibres from 2 to 5 mm long and having a diameter of from 1 to 5  $\mu$ , and had a surface area of 2.5 sq m/g. 15 g of these fibres were admixed with 350 g of cellulose (60% Husum Birch, 20% Modo Crown and 20% Husum Kraft) in 25 litres of water, and immediately dispersed in the water. Following the procedures of Example 1, from this paste there were prepared sheets whose properties are in Table 7.

#### Example 20.

Into the autoclave of Example 17, there were charged 6 kg of a polyethylene as in Example 17, 30 g of a C<sub>10</sub>-C<sub>12</sub> alcohol ethoxylated with ethylene oxide (molar ratio of ethoxylation=1:2) surfactant, and 70 litres of technical hexane. By heating with oil, there were established in the autoclave a temperature=172° C; a total pressure=12 kg/sq cm; and a nitrogen overpressure=3.5 kg/sq cm. Using the same procedure and equipment as in Example 17, but with the nozzles arranged at an angle of 65°, the polyethylene solution was ejected and hit, at a distance of about 3 mm from the nozzle, by a nitrogen current at room temperature. The temperature of the solution=190° C; the diameter of solution-ejecting nozzle=2 mm; the flow rate of the solution=105 kg/h; the pressure of the solution at the nozzle=12 kg/sq cm; the diameter of the nitrogen-ejecting nozzle=4 mm; the pressure of the nitrogen at the nozzle=21 kg/sq cm; and the impact speed of the nitrogen=320 m/s. The product consisted completely of fibres from 1 to 3 mm long with a diameter of from 1 to 20  $\mu$ , and had a surface area of 4.5 sq m/g. 150 g of these fibres were kneaded together with 350 g of cellulose (60% Husum Birch, 20% Husum Kraft and 20% Modo Crown) in 25 litres of water. Complete dispersion was instantaneous. Following the procedures described in Example 1, with this paste were prepared sheets whose properties are in Table 7.

#### Example 21.

Into the autoclave of Example 17, there were charged 7 kg of a polyethylene as in Example 17, 3 kg of a calcined clay with 95% of its particles below 10  $\mu$ ; 35 g of a surfactant condensation product of one mol of stearic



acid with 5.5 mols of ethylene oxide, and 80 litres of technical hexane. By heating, there were established inside the autoclave a temperature=148° C; a total pressure=7.7 kg/sq cm; and a nitrogen overpressure=2.2 kg/sq cm. Using the same procedures and equipment as in Example 17, but with the nozzles arranged at an angle of 55°, the mixture containing polyethylene in solution was ejected into the ambient atmosphere through a nozzle and hit at a distance of about 4 mm by an oxygen current at room temperature. The temperature of the solution at the nozzle=151° C; the diameter of the solution-ejecting nozzle=2 mm; the flow rate of the solution=70 kg/h; the pressure of the solution at the nozzle=6 kg/sq cm; the diameter of the oxygen-ejecting nozzle=4 mm; the pressure of the oxygen at the nozzle=21 kg/sq cm; and the impact speed of the oxygen=320 m/s. The product consisted of 8% of fibres of from 3 to 5 mm long and with a diameter of from 1 to 5  $\mu$ , and of 20% of flat fibres from 3 to 5 mm long, from 20 to 50  $\mu$  wide and from 1 to 5  $\mu$  thick, and had a surface area of 2.5 sq m/g, and a Specific Gravity (at 23° C) of 1.163. 150 g of these fibres were kneaded together with 350 g of cellulose (60% Husum Birch, 20% Husum Kraft and 20% Modo Crown) in 25 litres of water. Complete dispersion followed immediately. Using this paste, with the procedures of Example 1, sheets were prepared and their properties are in Table 7.

#### Example 22.

Into the autoclave of Example 17, there were charged 7 kg of polyethylene as in Example 17, 3 kg of clay as in Example 21, 35 g of a surfactant monolauric ester of sorbitol, and 80 litres of technical hexane. By heating there were established in the autoclave a temperature=147° C; a total pressure=8.7 kg/sq cm; and a nitrogen over pressure=3.5 kg/sq cm. Using the procedures and the equipment of Example 17, but with the nozzles arranged at an angle of 70°, the mixture containing the polyethylene in solution was conveyed to the nozzle and ejected into the ambient atmosphere, where the jet was hit, at a distance of about 4 mm from the nozzle, by an oxygen flow at room temperature. The temperature of the solution at the nozzle=165° C; the diameter of the solution-ejecting nozzle=2 mm; the flow rate of the solution=60 kg/h; the pressure of the solution at the nozzle=8.3 kg/sq cm; the diameter of the oxygen-ejecting nozzle=4 mm; the pressure of the oxygen at the nozzle=20 kg/sq cm; and the impact speed of the oxygen=320 m/s. The product consisted of 70% of fibres of from 1 to 5 mm long and having a diameter of from 1 to 20  $\mu$  and of 30% of flat fibres from 1 to 5 mm long, from 20 to 50  $\mu$  wide and from 1 to 20  $\mu$  thick,

and had a surface area of 2.5 sq m/g and a density (at 23° C) of 1.166 g/cc. A mixture of 150 g of these fibres with 350 g of cellulose (60% Husum Birch, 20% Husum Kraft and 20% Modo Crown) was kneaded in 25 litres of water. Complete dispersion followed immediately. With this paste and following the procedures of Example 1, sheets were prepared whose properties are in Table 7.

#### Example 23.

Into the autoclave of Example 17, there were charged 7 kg of polyethylene as in Example 17, 3 kg of calcined clay as in Example 21, 35 g of surfactant C<sub>10</sub>-C<sub>12</sub> alcohol ethoxylated with ethylene oxide (molar ratio of ethoxylation=1:5), and 80 litres of technical hexane. By heating, there were established in the autoclave a temperature=169° C; a total pressure=10.9 kg/sq cm; and a nitrogen overpressure=2.8 kg/sq cm. Through a pipe sheathed with a steam-heated sleeve, the mixture was ejected into the ambient atmosphere through a nozzle, and the jet was hit at a distance of about 2.5 mm from the ejection nozzle by a saturated steam jet coming out of a nozzle arranged at an angle of 85° to the first nozzle. The temperature of the solution at the nozzle=180° C; the diameter of the solution-ejecting nozzle=2 mm; the flow rate of the solution=105 kg/h; the pressure of the solution at the nozzle=11.5 kg/sq cm; the diameter of the steam-ejecting nozzle=4 mm; the impact of the steam=450 m/s. The product consisted of 90% of fibres from 2 to 5 mm long and with a diameter of from 1 to 5  $\mu$ , and of 10% of flat fibres from 2 to 5 mm long, from 20 to 50  $\mu$  wide and from 1 to 5  $\mu$  thick and had a density (at 23° C) of 1.168 g/cu.cm. A mixture of 150 g of these fibres with 350 g of cellulose (60% Husum Birch, 20% Husum Kraft and 20% Modo Crown) was kneaded with 25 litres of water. Immediate and completely homogeneous dispersion was obtained. Using this paste and operating according to the procedures of Example 1, sheets were prepared whose properties are in Table 7.

#### Example 24.

Into a 50 litre autoclave provided with a heating sleeve and a stirrer, there were charged 1.4 kg of a polyethylene prepared using an unsupported Ziegler catalyst and having a melt index=18; Specific Gravity=0.9630; number of methyl groups per hundred C atoms=0.26; and melt temperature (DSC)=133° C, together with 0.6 kg of a ground calcium carbonate (90% of the particles below 10  $\mu$ ), 40 g of a surfactant alkylphenol ethoxylated with 4 mols of ethylene oxide, and 14 litres of technical hexane. The mixture was heated in the autoclave by sending steam through the sleeve until a temperature=150° C; and a pressure=5.4 kg/sq cm were



obtained. The mixture containing the polyethylene in solution was ejected through a nozzle of 2 mm diameter into the ambient atmosphere and hit, at a distance of about 5 mm from the nozzle, by the flow of saturated steam ejected from a nozzle of 4 mm diameter, arranged at an angle to the first nozzle of about 90°. The flow rate of solution=15 kg/h; and the impact speed of the steam=420 m/s. The product consisted of 70% of fibres from 1 to 3 mm long and with a diameter of from 1 to 15  $\mu$ , and of 30% of flat fibres from 1 to 3 mm long, from 50 to 100  $\mu$  wide and from 1 to 15  $\mu$  thick, and contained less than 0.3% of solvent, and had a density (at 23° C) of 1.162 g/cu.cm. A mixture consisting of 150 g of these fibres and of 350 g of cellulose (60% Husum Birch, 20% Husum Kraft and 20% Modo Crown) was kneaded with 25 litres of water. Instantaneous dispersion in the water of the fibrous mixture followed. With the paste, and operating according to the procedures of Example 1, sheets were prepared whose properties are in Table 7.

#### Example 25.

This example illustrates the preparation of fibres starting from a polyethylene solution free from surfactants, and a comparison is made between the dispersability in water of the fibres thus obtained and that of the fibres prepared in the presence of a surfactant, according to the preceding Examples.

Into the autoclave of Example 24 there were charged 2 kg of polyethylene as in that Example, 0.260 kg of talc and 20 litres of technical hexane. By heating, there were established in the autoclave a temperature=152° C; and a pressure=5.2 kg/sq cm. The mixture containing the polyethylene in solution was ejected into the ambient atmosphere through a nozzle of a diameter of 2 mm, and was hit at a distance of 1.5 mm with a jet of carbon dioxide ejected by a nozzle of 4 mm diameter at an angle of 90°. The flow rate of the solution=15 kg/h; and the impact speed of the carbon dioxide=450 m/s. The product consisted of 70% of fibres from 1 to 2 mm long and from 1 to 20  $\mu$  diameter, and of 30% of flat fibres from 1 to 2 mm long, from 50 to 100  $\mu$  wide and from 1 to 20  $\mu$  thick, and had a density (at 23° C) of 1.050 g/cu.cm. 150 g of the fibres thus obtained and 350 g of cellulose (60% Husum Birch, 20% Husum Kraft and 20% Modo Crown) were mixed together in 25 litres of water. After 5 minutes a good dispersion was obtained. The paste was processed according to Example 1. The properties of the paper are in Table 7.

#### Example 26.

Into the same autoclave of Example 17, there were charged 7 kg of a polyethylene as in Example 17, together with 20 g of a

surfactant ethoxylated stearylamine, 80 litres of technical hexane and 3 kg of the calcined clay as in Example 21. By heating, there were established in the autoclave: a temperature=169° C; a total pressure=10.9 kg/sq cm; and a nitrogen over-pressure=2.8 kg/sq cm. Through a pipe sheathed with a steam heated sleeve, the mixture containing the dissolved polyethylene was conveyed to the nozzle and ejected into the ambient atmosphere, where it was hit at a distance of 2 mm from the nozzle and at right angle by a saturated steam jet. The temperature of the solution at the nozzle=180° C; the diameter of the solution-ejecting nozzle=2 mm; the flow rate of the solution=150 kg/h; the pressure of the solution at the nozzle=11.5 kg/sq cm; the diameter of steam-ejecting nozzle=4 mm; the impact speed of the steam=450 m/s. The product consisted of 70% of fibres from 1 to 3 mm long and with a diameter of from 1 to 15  $\mu$ , and of 30% of flat fibres having a length of from 1 to 3 mm, a width of from 20 to 50  $\mu$  and a thickness of from 1 to 15  $\mu$ . The density (at 23° C) of the product was 1.166 g/cc. A mixture of the fibres thus obtained and 350 g of cellulose (60% Husum Birch, 20% Husum Kraft and 20% Modo Crown) was kneaded with 25 litres of water. To obtain an immediate homogeneous dispersion. Using the paste and operating according to Example 1, sheets were prepared whose properties are in Table 7.

The following Examples illustrate one of the preferred embodiments of the invention: using the hitting fluid in the form of a mass geometrically coaxial with the ejecting-solution nozzle.

#### Example 27.

Into a 20 litre stainless steel autoclave, fitted with a heating sleeve and provided with a blade stirrer, these were charged 800 g of polyethylene, obtained using a Ziegler catalyst, non-modified monostadium, and having melt index=1.6; Specific Gravity=0.9525; number of methyl groups per 100 C atoms less than 0.1; melt temperature (DSC)=133° C, were introduced with 6 g of a surfactant ethoxylated stearylamine and 10 litres of technical hexane. By oil heating, there were established in the autoclave a temperature=185° C; a total pressure=13.0 kg/sq cm; and a nitrogen overpressure=3.5 kg/sq cm. Monostadium polyethylene is prepared in a single polymerization stage in order to obtain a low dispersion of molecular weights in the polymer.

A solution of polyethylene in hexane was thus obtained. For the preparation of the fibres starting from this solution, there was used a system of circular co-axial nozzles as in Figure 3 having a diameter of the solution ejecting nozzle 13=2 mm; a diameter of

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the hitting fluid ejecting nozzle 14=4 mm; length of chamber 15=10.4 mm; a maximum diameter of chamber 15=7.5 mm; and a value of angle  $\alpha=80^\circ$ . With reference to

5 Figure-3, through a thermally-insulated duct the polyethylene solution was fed into the duct 11, while saturated steam was fed into the duct 12. The flow rate of the solution=105  
10 kg/h; and the impact speed of the steam=210 m/s. The product gathered consisted of 90% of fibres from 4 to 5 mm long and having a diameter of from 1 to 5  $\mu$ , and of 10% of flat  
15 fibres from 4 to 5 mm long, from 15 to 20  $\mu$  wide and from 1 to 5  $\mu$  thick, and had a surface area of 4 sq m/g.

#### Example 28.

Into the autoclave of Example 27 there were introduced 900 g of polyethylene as in Example 27, and 10 litres of technical hexane.  
20 By oil heating there were established in the autoclave a temperature=170° C; a total pressure=11.9 kg/sq cm; and a nitrogen overpressure=3.5 kg/sq cm. A nozzle as in Example 27 was used but having an angle  
25  $\alpha=50^\circ$ . Using a duct sheathed by a sleeve and heated by steam, the duct 11 was fed with the polyethylene solution, while into the duct 12 there was fed a nitrogen flow. The temperature of the solution=190° C; the flow rate of  
30 the solution=95 kg/h; the impact speed of

the nitrogen=320 m/s. The product obtained consisted almost exclusively of fibres from 4 to 5 mm long and from 1 to 3  $\mu$  thick, and having a surface area of 3.5 sq m/g.

#### Example 29.

Into the autoclave of Example 27, there were introduced 720 g of polypropylene with a high index of syndiotacticity, obtained using a Ziegler catalyst and having a melt index=6.5; a Specific Gravity=0.9083; and a melt temperature (DSC)=160° C together with 6 g of a surfactant condensation product of 1 mol of stearic acid with 5.5 mols of ethylene oxide and 10 l of technical hexane.  
45 By heating, there were established in the autoclave a temperature=171° C; a total pressure=8.8 kg/sq cm; and a nitrogen overpressure=3.0 kg/sq cm. A circular nozzle system as in Example 27, but having an angle  $\alpha$  of 45° was used. Through a sleeved and steam-heated pipe, the duct 11 was fed with a polypropylene solution while into the duct 12 there was fed an oxygen flow. The temperature of the solution=190° C; the flow rate of the solution=90 kg/h; and the impact speed of the oxygen flow=420 m/s. The product consisted totally of fibres from 4 to 5 mm long, with a diameter of from 1 to 3  $\mu$ , and had a surface area of 4 sq m/g.

TABLE 1

	Degree of beating S.R.	Weight g/sq m	Thickness $\mu$	Breaking load in kg.	Elongation at break in %
RAUMA-type sulphite conifer cellulose	22	61	130	44	2.5
	27	64	130	43	2.5
	35.5	62	120	4.7	2.5
	41	62	120	5.5	2.0
Example 1	21.5	62	150-200	1.9	2.0
	27	61	140-200	2.5	2.0
	34	59	100-180	2.3	2.0
	42	57	140	2.5	2.5
Example 2	22.5	64	120-150	2.6	2.0
	34	63	120-140	2.6	1.5
	42	67	120-150	3.5	1.5
	52	67	120-130	3.5	1.5
Example 4	25.5	61	130	2.1	2.0
	34	58	120	2.5	2.0
	45	59	120	3.0	2.0

TABLE 1 (Continued)

	Length of break m	Bursting strength Kg/sq cm	Tearing in g	Porosity cu.cm/min
RAUMA-type sulphite conifer cellulose	4,800	1.5	54	1550
	4,500	1.7	55	920
	5,000	2.0	53	450
	5,900	2.1	50	320
Example 1	2,000	0.5	57	2050
	2,750	0.8	60	850
	2,600	0.9	60	450
	3,000	0.8	50	300
Example 2	2,700	0.9	70	1650
	2,750	1.0	70	650
	2,500	1.2	70	250
	2,500	1.2	64	100
Example 4	2,300	0.7	38	1000
	2,900	0.8	37	1150
	2,450	0.9	33	500

TABLE 2

	Rauma-Type Cellulose				70% Rauma 30% of Polyethylene Fibres				Modo-Type Fibres (Mooch Domsjos)			
	28	39	44	54	25	36	44	57	24	34	44	56
Degree of beating S.R.												
Weight, g/sq m	66.6	65.7	65.8	66.4	62.20	63.92	61.20	66.40	64.4	62.2	64.5	63.6
Breaking load KG	2.5	3.2	2.8	3.6	1.52	1.90	1.63	1.98	3.0	3.8	4.3	4.7
Elongation at break %	2.4	2.4	2.3	2.9	2.3	2.6	2.4	2.6	1.9	2.6	3.2	3.2
Length of breaking m	2500	3250	2850	3600	1659	2000	1800	200	3100	4000	4450	4900
Tear resistance g	112	92	104	96	72	80	72	64	104	84	84	64

TABLE 2 (Continued)

	70% Mado, 30% of Polyethylene fibres				Birch Tree cellulose				70% Birch, 30% Polyethylene fibres			
	25	35	45	50	25	33	43	58	22	29	36	47
Degree of beating S.R.												
Weight g/sq m	63.96	80.92	61.16	61.64	72.4	85.0	67.0	65.0	65.76	63.80	86.80	60.60
Breaking load Kg	1.97	1.66	2.06	2.56	2.5	3.5	4.9	4.8	1.63	2.04	2.17	2.23
Elongation at break %	2.8	1.8	2.0	2.9	2.2	2.5	3.2	3.4	2.3	2.4	2.9	2.7
Length of breaking m	2050	1800	2250	2750	2300	3550	4850	4900	1850	2150	2150	2500
Tear resistance g	84	74	72	72	60	80	80	60	52	68	60	56





TABLE 5

	Reference cellulose (60% Birch, 20% Modo, 20% Kraft)			Example 13	Example 14	Example 15
Degree of refining S.R.	26	38.5	47.5	56	37.6	37.7
Weight g/sq m	61	62	60	61	60	52
Breaking load kg	6.7	8	8	8	4.1	4.1
Elongation at break %	2.5	2.5	2.5	2.5	2.9	2.8
Length of break m	7320	8600	8830	8750	4670	5410
Tearing resistance g	41	44	40	38	38	20

TABLE 6

Test	A	B	C
Weight g/sq m	70.4	68.1	69.5
Longitudinal breaking load in kg	6.6	3.51	4.8
Transversal breaking load in kg	2.04	1.35	1.8
Length of longitudinal break m	6300	3440	4250
Length of transversal break m	1930	1350	1800
Average length of break in m	4110	2400	3020
Elongation at longitudinal break in %	3.1	3.0	3.0
Elongation at transversal break in %	5.3	3.8	3.9



TABLE 7

Example	17	18	19	20	21	22	23	24	25	26
Degree of beating SR at 22°C	41	45.5	40	37.5	35	43	33	38	38.5	38
Weight g/sq m	63.8	64	65.5	66.6	62.5	64.3	65.9	62.4	64.4	63.8
Breaking load kg	5.35	5.09	5.61	4.20	4.72	4.62	5.33	4.08	5.56	4.70
Elongation at break %	2.3	2.05	2.4	1.9	2.3	2.0	2.9	2.2	2.3	2.0
Length of break m	5258	4962	5431	4026	4980	4485	4871	4087	5180	4595

## WHAT WE CLAIM IS:—

1. A process of producing olefin polymeric fibres suitable for use in pulp in the manufacture of paper which comprises ejecting a solution of an olefin polymer as a jet, and hitting the jet of solution with a jet of fluid.
2. A process according to claim 1 in which the jets are inclined to each other at an angle of from 30° to 90°.
3. A process according to claim 1 or 2 wherein the olefin polymer is polyethylene.
4. A process according to claim 1 or 2 wherein the olefin polymer is a polyethylene containing a minor proportion of copolymerized units of another monomer.
5. A process according to claim 1 or 2 wherein the olefin polymer is a polypropylene consisting essentially of isotactic macromolecules.
6. A process according to claim 1 or 2 wherein the polymer solution includes a polymer containing polar groups.
7. A process according to any of the preceding claims wherein the speed upon impact of the fluid jet is from 200 to 600 m/s.
8. A process according to any of the preceding claims wherein the fluid is steam.
9. A process according to any of claims 1 to 7 wherein the fluid is carbon dioxide.
10. A process according to claim 8 or 9 wherein the angle between the direction of the fluid and that of ejection of the solution is from 80° to 85°.
11. A process according to any of claims 1 to 7 wherein the fluid is nitrogen.
12. A process according to claim 11 wherein the angle between the direction of the fluid and that of ejection of the solution is from 50° to 55°.
13. A process according to any of claims 1 to 7 wherein the fluid is oxygen.
14. A process according to claim 13 wherein the angle between the direction of the fluid and that of ejection of the solution is from 40° to 60°.
15. A process according to any of the pre-

- ceding claims in which the solution is ejected into the ambient atmosphere.
16. A process according to any of the preceding claims wherein the olefin polymeric solution contains a surfactant.
17. A process according to any of the preceding claims wherein the fluid is in the form of a mass which is geometrically co-axial with the nozzle ejecting the olefin polymeric solution.
18. A process according to any of the preceding claims wherein the temperature of the solution is at least 60° C higher than the boiling temperature of the solvent under normal conditions.
19. A process according to any of the preceding claims wherein the solvent is hexane.
20. A process according to any of claims 1 to 18 wherein the solvent is heptane.
21. A process according to any of claims 1 to 18 wherein the solvent is trichlorofluoromethane.
22. A process according to any of the preceding claims wherein the olefin polymeric solution is ejected at a speed of from 1,000 to 200,000 m/h.
23. A process of producing olefin polymeric fibres as described in any of Examples 1 to 4.
24. A process of producing olefin polymeric fibres as described in Example 5.
25. A process of producing olefin polymeric fibres as described in Example 6 or 7.
26. A process of producing olefin polymeric fibres as described in Example 8.
27. A process of producing olefin polymeric fibres as described in any of Examples 9 to 12.
28. A process of producing olefin polymeric fibres as described in any of Examples 13 to 15.
29. A process of producing olefin polymeric fibres as described in Example 16.
30. A process of producing olefin polymeric fibres as described in any of Examples 17 to 26.
31. A process of producing olefin polymeric fibres as described in any of Examples 27 to 29.
32. Fibres produced by a process according to any of the preceding claims.
33. Paper containing fibres according to claim 32.
- ERIC POTTER AND CLARKSON,  
Chartered Patent Agents,  
25 The Crescent,  
Leicester.

1392667

COMPLETE SPECIFICATION

4 SHEETS

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Sheet 1

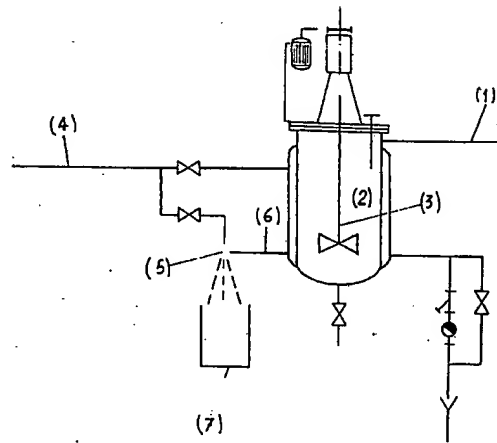


FIG. 1

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Sheet 2

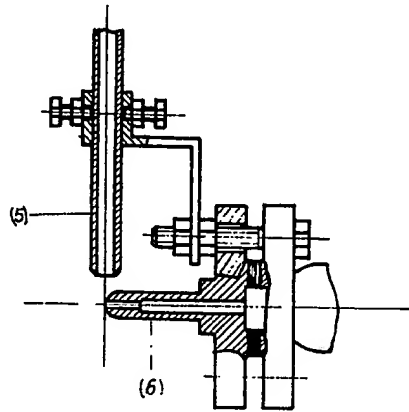
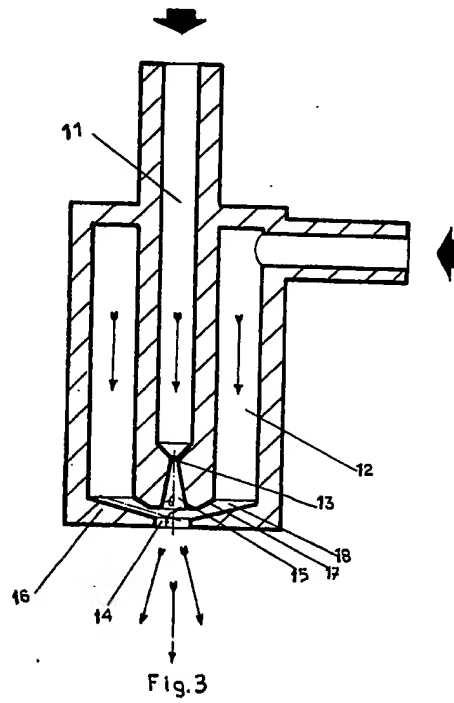


Fig 2



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COMPLETE SPECIFICATION

4 SHEETS

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Sheet 4

